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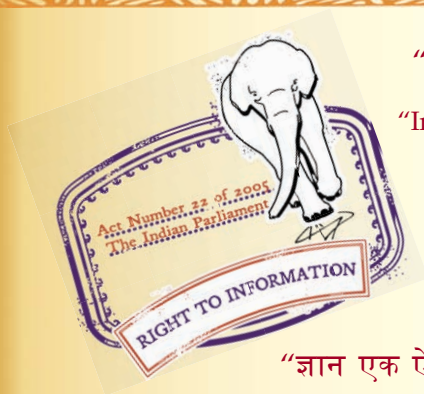
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“Step Out From the Old to the New”

IS 12667-4 (1989): Methods of chemical analysis of chromite sand, Part 4: Determination of calcium [MTD 13: Ores and Raw Materials]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

**CHROMITE SAND FOR FOUNDRIES —
METHODS OF CHEMICAL ANALYSIS**

PART 4 DETERMINATION OF CALCIUM

भारतीय मानक

फाउंट्रियों के लिए क्रोमाइट सैंड — रासायनिक विश्लेषण

भाग 4 कैल्सियम ज्ञात करना

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FOREWORD

This Indian Standard (Part 4) was adopted by the Bureau of Indian Standards on 29 March 1989, after the draft finalized by the Methods of Chemical Analysis of Ores, Minerals and Refractories Sectional Committee had been approved by the Structural and Metals Division Council.

IS 6788 : 1973 'Specification for chromite sand for foundries' covers the requirements for the various constituents. As chromite sand is widely used in steel/iron castings, a need was felt to prepare the standard on methods of test based on the requirements of IS 6788 : 1973.

This standard has been prepared in different parts. This part (Part 4) covers the chemical analysis of calcium, and other parts are:

Part 1 Determination of silica

Part 2 Determination of iron

Part 3 Determination of chromium

In reporting the results of test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

CHROMITE SAND FOR FOUNDRIES — METHODS OF CHEMICAL ANALYSIS

PART 4 DETERMINATION OF CALCIUM

1 SCOPE

1.1 This standard (Part 4) covers a method for determination of calcium (as CaO) in chromite sand for foundries.

2 REFERENCES

2.1 The Indian Standards listed below are necessary adjuncts to this standard:

- IS 264 : 1976 Specification for nitric acid (second revision)
IS 265 : 1987 Specification for hydrochloric acid (third revision)
IS 1811 : 1984 Methods of sampling foundry sand (first revision)

3 SAMPLING

3.1 The sample shall be drawn by the method prescribed in IS 1811 : 1984.

3.2 Pulverize about 10 g of sample in an agate mortar to pass through 75 μ m IS test sieve. Dry at 105 to 110°C for 2 hours.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, analytical grade reagents and deionized water shall be employed in the test.

5 DETERMINATION OF CALCIUM BY ATOMIC ABSORPTION SPECTROPHOTOMETRIC (AAS) METHOD

5.1 Out line of the Method

The sample is dissolved in hydrofluoric acid and nitric acid mixture and calcium is determined by AAS at 422.7 nm.

5.2 Apparatus

Atomic absorption spectrophotometer capable of reading absorbance in the region of 422.7 nm and equipped with nitrous oxide-acetylene burner.

5.3 Reagents

5.3.1 Concentrated Hydrochloric Acid, rd = 1.16 (conforming to IS 265 : 1987)

5.3.2 Concentrated Nitric Acid, rd = 1.42 (conforming to IS 264 : 1976).

5.3.3 Perchloric Acid, 70 percent.

5.3.4 Hydrofluoric Acid, 40 percent.

5.3.5 Potassium Bromide Solution, Dissolve 30.5 g of potassium bromide in 1 litre of water.

5.3.6 Standard Calcium Solution

Dissolve 0.624 g of pure calcium carbonate in 25 ml of dilute hydrochloric acid (1:1). Transfer to 250 ml volumetric flask, dilute with water and mix. Store in a polyethylene bottle. Transfer 25 ml of the above solution to 250 ml volumetric flask, dilute with water and mix (1 ml = 0.1 mg Ca).

5.4 Procedure

5.4.1 Weigh 0.500 g of sample (see 3.2) into polytetrafluoroethylene beaker of 75 ml. Add 20 ml of concentrated hydrochloric acid, 1 ml of concentrated nitric acid and 10 ml of perchloric acid. Digest the contents till a syrupy mass appears. Add 5 ml more of perchloric acid, 1 to 2 drops of hydrofluoric acid and fume to syrupy mass. Cool and add 10 ml dilute hydrochloric acid. Transfer the solution to 100 ml volumetric flask. Add 10 ml potassium bromide solution and make up.

5.4.2 Blank

Prepare a reagent blank using the same quantity of reagents.

5.4.3 Adjustment of Atomic Absorption Spectrometer

Set the wave length for calcium at 422.7 nm. Fit the burner in accordance with the manufacturer's instruction and light the nitrous oxide-acetylene flame. After 10 minutes pre-heating of burner, optimize the instrumental parameters in accordance with the instructions given in the manual.

Aspirate water and calibration solution to establish that the absorbance reading is not drifting and then set the initial reading for water to zero absorbance.

5.4.4 Atomic Absorption Measurements

Aspirate the calibration solution and final test solution in order of increasing absorption starting with blank solution, with the final test solution being aspirated at a proper point in the series. Aspirate water between each solution and record the reading when stable responses are obtained. Repeat the measurements once more and average the readings.

5.4.5 Calibration Curve

Take 0, 5, 10, 15, 20 and 25 ml of standard calcium solution (*see* 5.3.6) and proceed according to the procedure mentioned above. Prepare a calibration curve by plotting the net absorbance values of standard solution against

the concentration of calcium (as mg/ml). Convert the net absorbance values of the test solution to milligrams of calcium by means of calibration curve.

5.5 Calculation

Calculate the percentage of calcium as (CaO) as follows:

$$\text{Calcium (as CaO), percent} = \frac{A}{B} \times \frac{1}{10} \times 1.4$$

where

A = mg of calcium/ml, and

B = g of sample represented in 100 ml volume of final test solution.

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